THE STUDY OF ELECTROSPUN NANOFIBERS AND THE APPLICATION OF ELECTROSPINNING IN ENGINEERING EDUCATION

A Thesis

by

CHRISTOPHER CALVIN CALL

Submitted to the Office of Graduate Studies of
Texas A&M University
in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE

August 2008

Major Subject: Mechanical Engineering

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Approved by:

Chair of Committee,	Christian Schwartz
Committee Members,	Zoubeida Ounaies
	Terry Creasy
Head of Department,	Dennis O'Neal

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ABSTRACT

The Study of Electrospun Nanofibers and the Application of Electrospinning in Engineering Education. (August 2008).

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Chair of Advisory Committee: Dr. Christian Schwartz

During electrospinning, a polymer solution becomes an electrically driven jet as it travels to a grounded plate. While the behavior of pressure-driven liquid jets has been extensively studied in fluid mechanics, none of the characteristics of fluid jet break up have been applied to electrospinning. Calculating Weber number can describe what type of breakup occurs as the polymer jet travels to the plate, which could also predict the surface morphology of electrospun fibers. Polyethylene oxide (PEO) solution was electrospun at different voltages to test whether the morphology of the electrospun fibers can be predicted through calculating Weber number. While the continuing research of electrospinning is important, the subject of electrospinning can be used as a course to teach students engineering principals over a semester. Due to the vast interdisciplinary subjects associated with electrospinning, teaching the subject as a course will give students an understanding of critical thinking skills as well as first hand accounts of research.

Four weight percent PEO solution was electrospun at a range of testing parameters until the desired results were achieved, beaded or non-beaded fibers. The Weber numbers were calculated and compared to the electrospun material created. Analyzing the surface morphology revealed a beaded to non-beaded trend in nanofibers corresponding to high-to-low Weber numbers. The same trend continued for higher weight percents of PEO solutions electrospun.

The course will have many learning objectives the instructor is expected to have the students achieve, building the objectives to help the students become better researchers and to learn the material. Splitting the course into three five week sections will help students understand each component of the electrospinning process, as well as fundamental engineering equations and theories. The students at the end of the semester should be able to recreate the electrospinning process on their own and create nanofibers of varying sizes. The course should also excite students about pursuing more advanced degrees in scientific fields.

DEDICATION

I dedicate my thesis to my family and friends. Without their support and encouragement, I would not be the person I am today nor at the place that I am now.

ACKNOWLEDGEMENTS

I would like to thank my committee chair, Dr. Schwartz, and my committee members, Dr. Ounaies and Dr. Creasy, for their guidance and support throughout the course of this research.

Thanks also go to my friends and colleagues and the department faculty and staff for making my time at Texas A&M University an experience. I want to give a big thank you to Kevin Plumlee for reading through my thesis multiple times over and making sure I had no big errors. I also want to thank the people of the Microscopy center for use of their equipment facilities and the student's in Dr Ounaies' lab for the use of their optical microscope.

NOMENCLATURE

PEO	Poly(ethylene oxide)
Wt %	Weight Percent
V	Volt
T	Time
SEM	Scanning Electron Microscope
g	Grams
mm	Millimeters
nm	Nanometers
um	micrometers
ml	milliliter
NTEO	New Taxonomy of Educational Objectives
I/P	Current/Pressure

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1. INTRODUCTION

Nanotechnology uses material that has dimensions on the scale of 10^{-9}m, approximately one thousand times smaller than the width of a single strand of human hair. Creating nano-materials gets scientists closer to controlling objects on the atomic scale. 10 Angstroms (Å), a common term used in explaining atomic sizes, is equivalent to 1 nm. The idea of controlling material on the nano-level has been a topic of much research in recent years. Material on the nanoscale has a great deal of potential in numerous applications. The main research focus of nanotechnology is currently directed towards biology, engineering disciplines, and space applications. While there are other areas of study that nanotechnology will greatly benefit, it is still a new area of research that is not completely understood.

One of the more popular processes to create nanofibers, a form of nanotechnology, is through electrospinning. Electrospinning is the creation of nanofibers by application of a very high electric field. Patented in the 1930's by Antonin Formhals, electrospinning offers great rewards in the creation of nanofibers [1], because it can consistently create materials on the nanoscale. Research in the electrospinning field has increased greatly in recent years due to the emergence of nanotechnology. Many of the studies done in electrospinning are performed to figure out how to make different polymers on the nanoscale and what processing parameters control the electrospinning output. Even with all of the discoveries made by scientist towards electrospinning, the process still seems more of an art than science.

This thesis follows the style of Polymer.

Another unique aspect of electrospinning is its potential to be taught in a college classroom. Teaching an emerging technology to a group of students would be a great challenge. However, electrospinning involves a lot of interdisciplinary concepts that would help reinforce many of the engineering principles taught in schools now, concepts including physics, determining the force created by an electrical charge, and chemistry, dealing with liquid solutions and chemical bonds. The multiple disciplines involved with electrospinning would give students an opportunity to see a real world application of their calculations and theories. Instead of the theoretical springs, gears, velocities, and other material used as examples to explain the principles of physics, chemistry, fluid dynamics, thermodynamics, and dynamics and kinematics. These hands-on examples are a rarity in learning and could help increase a student's interest in studying nanotechnology.

Whether it is through teaching or researching to improve technology, electrospinning is a unique process; one that should be understood by scientist as well as a teaching tool for students.

2. LITERATURE REVIEW

Electrospinning is the electrically driven drawing of a polymer solution to produce a polymer fiber on the sub-micron scale. Electrospinning is a fairly straightforward process to setup. The main components of an electrospinning system includes: a high voltage power supply, a dissolved polymer in a reservoir with a conductive nozzle, and an electrically grounded plate set a certain distance from the conductive nozzle.

The applications for electrospun fibers are vast and varied. In the biomedical field, they have been studied for wound dressing, tissue engineering, and in protective clothing [2-6]. In tissue engineering, electrospun fibers have a significant capacity to mimic the properties of the extracellular matrix within the body. Electrospun nanofibers also allow researchers to investigate cell attachment, differentiation, and material affects on a very small scale [2]. In wound dressing nanofibers are potentially useful because of their extensive surface area and small pore size, making infection less likely [2]. Other applications that electrospinning is well-suited for are the creation of reinforcing fibers in composite materials, insecticide on plants, and a non-wetting surfaces on textile materials [7].

Since the first patent in 1934 by Antonin Formhals [1], approximately 60 patents have been filed on different forms and processes of electrospinning. Before the recent resurgence of research done on electrospinning, due to the dawn of nanotechnology, most of the earlier studies that are now associated with electrospinning were done in other fields of study. Taylor discovered that when applying high electric forces to a polymer solution, the exposed droplet takes a conical shape and becomes an electrically driven jet [8]. This geometric characteristic of the droplet at the point of extrusion is referred to as the Taylor cone. It used today in many experiments as an indication of the onset of electrospinning. Once researchers started devoting more attention to making nanoscale materials, it was found that electrospinning was able to consistently make material on the sub-micron scale.

As more and more researchers began to use electrospinning to produce nanofibers, they began to note the presence of entrained beads along the length of the fiber after deposition on the collector plate. This phenomenon has been attributed to a capillary effect of the solution as it travels to the plate [9, 10]. Though entrained beads are a very distinctive form of morphology compared to uniform fibers, which has had an absence of work to determine the cause, other than the passing mention of the capillary effect. It has been hypothesized that as the polymer solution jet travels, domains in the jet seek to minimize free surface energy by pulling together locally and thus forming entrained spheres or beads. Depending on the distance to the collector plate, the individual spheres may not completely separate before reaching the target plate, leaving a string and bead effect with the resulting fiber. Figure 1 shows a polymer solution that has beads embedded with the nanofibers created.

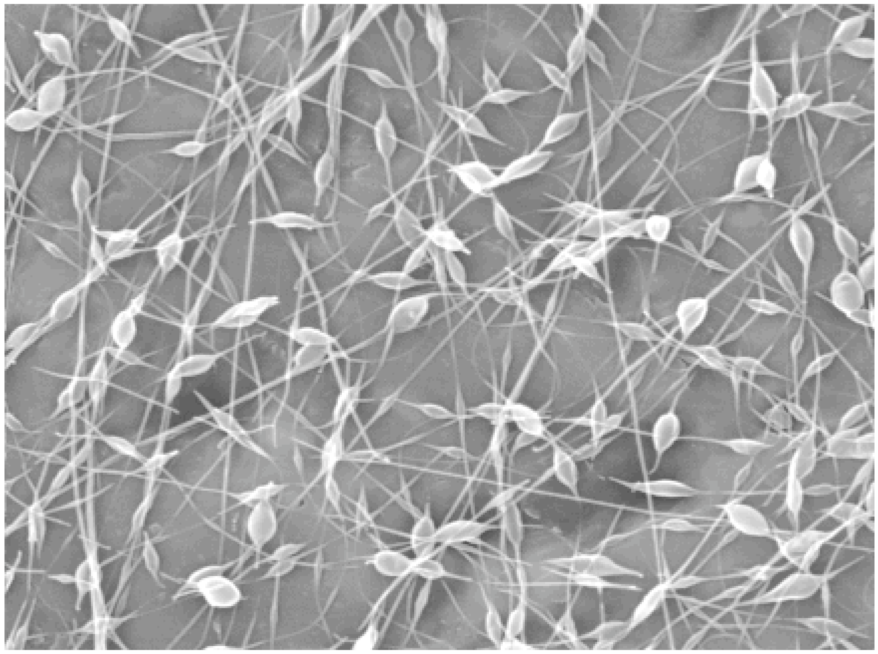


Figure 1: Beads created during the electrospinning process [11].

Fong *et al.* suggested that the beads were resultant of the high surface tension of the polymer solution [10]. High surface tension exacerbates the capillary effect described above. In Fong's study, it was shown that decreasing the surface tension of the solution will result in fewer beads formed, but the diameter of the fiber will be greater. Tripatanasuwan *et al.* accredits the bead formation to the capillary effect, but also shows that the humidity in the atmosphere can influence the evaporation rate of the polymer jet, thereby affecting the bead formation process [9]. By electrospinning in an enclosed environment, it was observed that as humidity was increased, so was the number of entrained beads in the fibers.

Tripatanasuwan *et al*. showed the humidity can affect the evaporation rate of a polymer solution; therefore, the surface tension of a polymer solution is not the only factor in the creation of beaded fibers. In Tripatanasuwan *et al*. paper certain factors such as: voltage, weight percent, density, and viscosity are held constant to prove humidity's affect on fiber morphology. However, by increasing the distance between plates, changing the velocity of the jet, and controlling the flow rate of the polymer solution, all can have an effect how well a solvent will evaporate when being electrospun.

Another significant discovery about the electrospinning process is the phenomenon known as whipping. While the polymer jet travels to the collection plate, the solvent in the solution begins to evaporate, leaving the solidified polymer. As this is occurring, the jet undergoes seemingly random high-speed flagellations in transit to the plate, referred to as whipping. Many researchers have tried to make models explaining why the whipping phenomenon takes place and how to predict the erratic circular motion the jet will have when it is being electrospun [12, 13]. In Yarin *et al.*, an attempt is made to predict the amount of whipping that will occur by observing the disturbances caused by the electric force and the disturbances caused by the air. In this model, Yarin stated that the evaporation of the solvent and solidification of the polymer jet had a great effect on the radius of the circular pattern that is created when the fiber begins to whip. In the work of Reneker *et al.,* it was suggested that the whipping motion is caused by the opposing charges carried by the liquid jet, as it got closer to the plate. They also observed that as the whipping of the jet becomes greater, the diameter of the nanofibers becomes smaller [13]. Another group, Hohmann *et al.*, concluded that the whipping of a polymer jet is due to the interplay of electrical charges, gravitational forces, and

Rayleigh instability [14]. Figure 2 shows the whipping effect of a polymer solution as it travels to the collection plate.

Hohmann *et al*. paper gives a more descriptive view of the whipping phenomenon by attributing more variables: electrical, gravitational, and Rayleigh instability, into the causes of the whipping phenomenon, more than Reneker *et al*. and Yarin *et al.* do in their respective papers. Hohmann *et al* also goes into further detail by describing how electrospinning can be utilized with different viscosities and charge densities of polymer solution, by expressing a formula as a function of the two parameters to describe the electrospinning process [15].

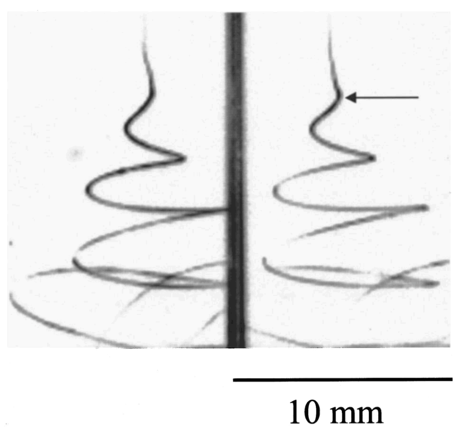


Figure 2: Illustration of the whipping effect in a polymer jet [3].

After discoveries of bead formation and whipping of the polymer jet, continued research in the electrospinning process has led to a variety of different polymers and solvents that can be used to create nanofibers. Huang *et al.* reported a detailed list of over forty-four different polymers that were successfully electrospun, along with the solvent used, testing parameters, applications for the specific polymer nanofibers, and the concentration of the polymer in the solution [16].

As further research continued, it was found that voltage and weight percent of the solution influenced the output of the fibers. Scientists have been able to further expand the list of different parameters that affect the final outcome of the electrospinning process. These factors include surface tension, distance between needle tip and collection plate, temperature, humidity, rate of fluid ejection, and polymer used [7, 11, 17-22]. A review of these results indicates that when the applied voltage is increased the diameter of the electrospun fibers becomes smaller, and the possibility of having entrained beads within the fibers increases, as well. Also, it has been reported that polymer solutions of high surface tension require a higher applied voltage to pull the fiber to the plate. Finally, increasing the distance between the nozzle and collector increases the possibility of pure fibers being spun, however a greater applied voltage is required.

One polymer that has been widely used in electrospinning is poly(ethylene oxide) (PEO). PEO is used in many biomedical applications such as drug delivery and tissue engineering [6, 22, 23]. It is preferred over other polymers because is very biocompatible and relatively simple to electrospin using multiple solvents and voltages [24]. Its easy polymer structure, two carbons and an oxygen molecule on the backbone, and ability to be dissolved in water make it fairly simple to create polymer solutions to electrospin material with. PEO is also readily combined with other polymers to produce composites for various applications [24].

Work by Dietzel *et al*. addresses details about process parameters specifically involved with the electrospinning of PEO nanofibers [22]. In this work, several electrospinning process parameters including the effects of voltage, surface tension, feed rate of solution, and the weight percent of polymer in the solution, are examined with regards to their effect on the fiber formation [22]. The paper shows that an increase in voltage leads to increases in the number of entrained beads in spun PEO nanofibers. It is also shown that with higher weight percent of PEO in the solution, the likelihood of bead formation is reduced.

Dietzel *et al*. does a very thorough job of explaining how different parameters can affect the electrospinning process. Dietzel et al. gives electrospinning results ranging from voltage applied, weight percent, flow rate, and the surface tension of the polymer solution, which describes a wider scope of the electrospinning process. Other papers neglect the parameters that affect electrospinning and are more focused on the characterization of resultant material as well as the potential applications [7, 17, 19, 21, 22]. Another paper that directly addresses some of the processing parameters affect on the fiber morphology is Huang *et al*. review of all the polymer created through electrospinning [16]. Huang *et al*. only mentions how voltage and viscosity affect the morphology of the electrospun material.

While it is easy to recognize the cause of the bead formation, the capillary effect of a polymer solution, the processing parameters used in electrospinning, voltage, distance traveled, and weight percent, are still being varied from polymer to polymer to avoid making beaded nanofibers. Because there is no clear-cut formula or material characteristic that dictates whether beads are created, control of fiber morphology is a challenge. One avenue that has not been explored thoroughly is using fluid mechanic principals to explain electrospinning morphology. Using fluid mechanics principal to describe the evolution and character of the polymer jet as it travels to the grounded collection plate may help in characterizing the resultant fiber morphology of the polymer nanofiber.

3. USING WEBER NUMBERS TO EXPLAIN SURFACE MORPHOLOGY OF ELECTROSPUN FIBERS

3.1 Introduction

Electrospinning is one of a few processes that have the capability to produce nanofibers. Electrospinning has potential in biomedical applications [25], fuel cells [26], filtration [27], and composites [28]. In biomedical applications, electrospinning can be used to control drug delivery rates that control therapeutic effects, convenience, and toxicity [23]. In filtration, electrospun mats can filter particles in the sub-micron range [25]. Both polymer and ceramic nanofibers can be electrospun to create fibers with diameters ranging from tens of nanometers to several microns.

Electrospinning is the electrically driven drawing of a polymer solution into a viscoelastic polymer jet that ultimately produces a mat of solid polymer fibers. In practical terms, the basic components of an electrospinning system include a high-voltage power supply, a polymer dissolved in solution, a chamber with a conductive nozzle to hold the solution, and a grounded collection plate that is separated from the nozzle by a chosen distance. By applying sufficient electric potential between the nozzle and the collection plate, a polymer jet is developed [8]. As the charged solution is pulled electrically to the collector plate, the diameter of the jet becomes smaller due to extensional flow and evaporation until a solidified polymer fiber is deposited on the plate. Depending on the chosen electrospinning parameters and the composition of the polymer solution, the morphology of the spun polymer can result in smooth or beaded fibers [10].

Many investigators have tried to explain the process through which fibers are created and determine the effect of electrospinning parameters on fiber morphology. Some parameters studied have been voltage, solution viscosity, solution surface tension, solution concentration, the distance between syringe needle and collection plate, temperature, solvents, and ambient humidity [10, 12, 18, 22, 25, 29, 30]. A number of tests have been done with voltages ranging from 5 kV to 30 kV and have been performed in atmospheric conditions. One characteristic behavior in fiber electrospinning is the production of a Taylor Cone of solution at the tip of the nozzle. This was first described by Lord Taylor in 1969 [8].

A challenge to investigators has been to fully understand the mechanics of the electrospinning process. Some have developed analytical models to aid in this pursuit. Yarin *et al.* proposed a mathematical model to predict how the polymer solution is pulled into a liquid jet and how the jet diameter decreases as the solution begins to spin [12]. This model also predicts where the fiber will be placed on the collection plate by considering the forces created by the electric field and by gravitational forces. This group was also able to model the characteristic whipping of the solution jet in a circular motion as it travels to the collector plate. Tripatanasuwan *et al.* went a step further in demonstrating and describing mechanistically what is happening to the jet as it travels to the plate [9]. They showed that humidity affects the evaporation rate of the solution, leading to beads entrained in the fibers and a smaller fiber diameter between the beads. This is referred to as the capillary effect [9], which occurs when the wavelengths of disturbances are greater than the diameter of the jet. These disturbances may be wind induced or electrical signals in the case of electrospinning polymer fibers [30]. In reaction to becoming unstable due to these disturbances, surface energy effects drive portions of the liquid jet to spherical shapes. During electrospinning, the solidification of the polymer takes place before separate spherical droplets can detach, thus resulting in a string of fibers with beaded "pearls" along the line. This is illustrated in Figure 3. While many electrospinning parameters (voltage, weight percent, solvent, distance traveled) have been shown to affect the fiber morphology of the electrospun polymer, there has been little substantive work to relate electrospinning to larger scale liquid jet phenomena, specifically those that involve fluid mechanics.

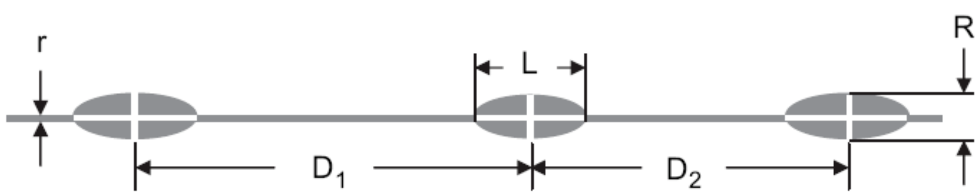


Figure 3: A look at the capillary effect of electrospun beaded fibers [9].

In this paper, the authors have focused on relating the mechanisms of fluid jet breakup to the morphology of electrospun nanofibers. Outside of the realm of electrospinning research, Lin *et al.* studied the types of jet breakup that can occur when a pure fluid (water) is propelled out of a nozzle, using material properties such as viscosity, surface tension, characteristic diameter, and velocity [30]. Lin was able to predict the distance that a fluid jet will travel before it begins to break up into a series of droplets by calculating the Weber number of the liquid jet. The formula for determining this dimensionless quantity is as follows:

$$We = \frac{\rho U^2 d}{\sigma} \tag{1}$$

where U is the velocity of the jet, d is the characteristic diameter of the jet, ρ is the density of the solution, and σ is the surface tension of the solution. Essentially, Weber number indicates the ratio of inertial energy to surface energy in a free jet flow. It is believed to be applicable to the electrospinning process because the solution travels as a free jet to the collection plate. Many of the parameters or processing variables important in electrospinning fibers can be accounted for by the use of Weber number. The surface tension and density of a polymer solution are determined by the weight percent of dissolved polymer. The voltage and distance between nozzle and collection plate affects the diameter of the fibers created. The velocity of the jet can be controlled by the force exerted to pull the jet to the plate, which is produced by the applied voltage. In Lin's study, Weber number was used to indicate the type of jet breakup that would occur in larger jets as the Weber number crossed threshold values. In this study, the authors characterized the velocity and diameter of the electrospun fibers under varying parameters to determine if Weber number maintained relevance at the jet sizes produced by electrospinning. The fundamental goal of the work was to determine if Weber number could be used to predict fiber morphology and flow characteristics.

Poly (ethylene oxide) (PEO) dissolved in water has been extensively studied for electrospinning applications because of its solubility and its various uses; however, researchers have observed that beaded morphologies commonly occur when the fraction of polymer in the solution is lowered to amounts nearing four weight percent. In many cases, well-formed fibers are produced at higher PEO concentrations. Little explanation has been given for the sudden appearance of the entrained-bead morphology at low polymer concentrations. This paper details the experimental work involved in determining the flow parameters during electrospinning as they relate to Weber number and fiber morphology in order to determine if this approach can explain the appearance of the beaded morphology at low concentrations. The goal was to determine whether a threshold Weber number exists so as to allow fiber morphology to be predicted and controlled not only in PEO solutions, but also in other polymer systems.

3.2 Experimental Procedure

3.2.1 Surface tension measurement

Poly(ethylene oxide) (Sigma/Aldrich) with a molecular weight of 400,000 g/mol (M_v) was used in this study. Solutions of distilled water with between four and eight weight percent of PEO were prepared in the laboratory by adding PEO powder to the water and using a magnetic stir bar to completely dissolve the polymer. The solutions were then taken to a contact angle analyzer (Edmund Optics FTA188 Video Tensiometer) to measure their surface tensions.

3.2.2 Electrospinning

Electrospinning was performed in atmospheric conditions, and a digital thermometer (DinoCapture) was used to monitor the temperature and humidity of the ambient environment.

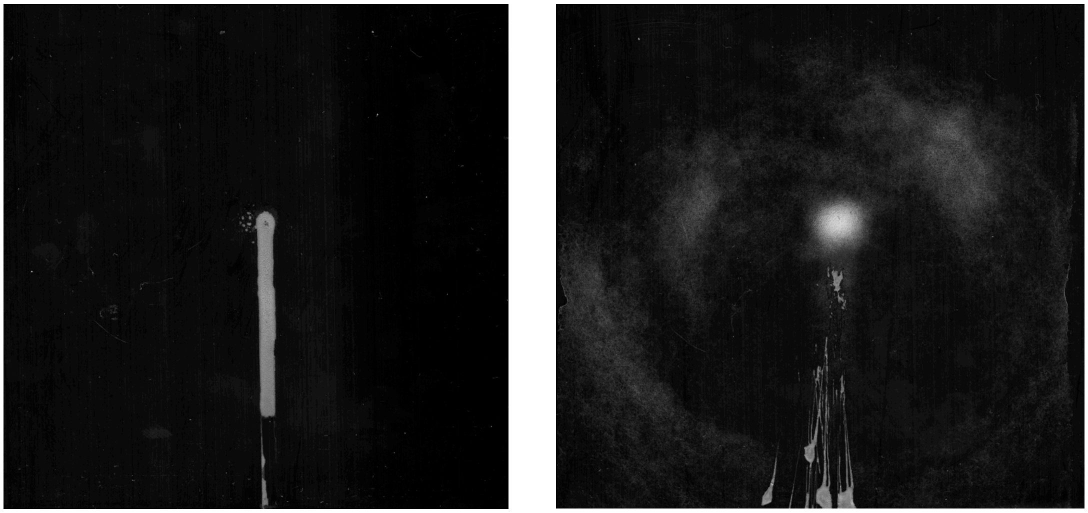
Initial test were performed to observe the polymer jet and placement of the resultant material while electrospinning. Initially, a pneumatic cylinder attached to current/pressure (I/P) transducer was used to eject the polymer solution from the syringe, making the testing variable pressure (Pa) instead of volumetric flow rate (ml/hr). One of the first things noticed in the initial test is that the resultant electrospun material would make different sized shapes or patterns on the collection plate, mostly circles. The look of the Taylor cone would also be different when different voltages were applied to the polymer solution. On higher voltages it looked as more solution was being pulled to the collection plate when compared to the solution being pulled to the plate at lower voltages, diameter of the liquid jet beginning from the needle tip.

A full factorial test to measure the differences and importance of the settings used in electrospinning was run. The settings being tested were voltage, pressure, weight percent, needle size, and molecular volume (M_v). The high and low settings for the full factorial can be seen in Table 1. The results of the factorial test performed were based on the weight of the PEO material electrospun with each test lasting 10 minutes.

Table 1: Full factorial test settings picked to electrospin the PEO solutions.

#	Setting	- (Low)	+ (High)
1	Voltage (kV)	7	10
2	Weight Percent (wt %)	4	8
3	Psia (Pa)	7	8
4	Needle Size	23	18
5	Molecular Volume (M_v)	400000	1000000

The results from the full factorial showed that all electrospinning done with the molecular volume of one million were too inconclusive with the processing parameters applied in the test. The one million M_v polymer solutions would have large amounts of liquid polymer reach the plate during testing, making it very difficult to record accurately the weight of electrospun material. The 18-gauge needle also proved an obstacle because the area of the needle was very large, causing more liquid to be pushed out then the voltages applied could accommodate. Liquid solution would build up on the tip of the needle and drop off the syringe to the counter top, or, in cases of the one million M_v, would travel to the plate as a liquid solution. Examples of both can be seen in Figure 4a and Figure 4b.



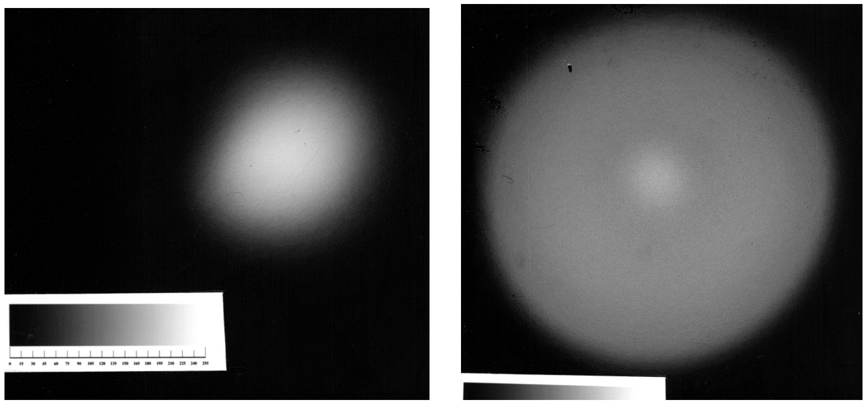
A) B)

Figure 4: Scanned results from full factorial test a) 4wt% PEO at 10kV b) 4wt% PEO at 7kV.

Another Factorial test was performed using only three factors: voltage, weight percent, and pressure, at high to low settings. The needle size was held constant at 23 gauge, M_v was at 400000, and each test lasted 10 minutes. The settings of the test and results can be seen in the Table 2 and Figure 5.

Table 2: Fractional factorial test setup and settings used.

#	+ (High)	- (Low)
Voltage (kV)	10	7
Weight Percent) (wt%)	8	4
Psia (Pa)	8	7
Needle Size	23 gauge needle	
Molecular Volume (M_v)	400,000	



A) B)

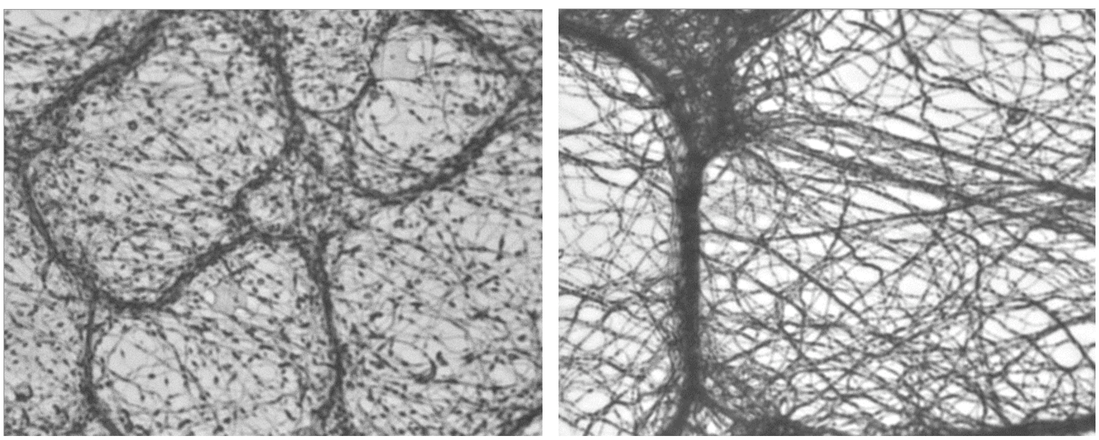
Figure 5: Images of electrospun material from fractional factorial test a) 8wt% PEO at 10kV b) 4wt% PEO at 10kV.

Once the test was completed, a 7-way analysis of variance, as seen in Table 3, was performed. The results from the test showed the weight percent and voltage were the two leading processing parameters involved in electrospinning.

Table 3: 7-way analysis of variance from fractional factorial test

SOURCE	DF	SUM OF SQUARES	MEAN SQUARE	F STATISTIC	F CDF	SIG
Total	31	0.000437	0.000014			
Voltage (V)	1	0.000055	0.000055	12.2326	99.82%	**
Weight Percent (WP)	1	0.000161	0.000161	35.6464	100%	**
Pressure (P)	1	0.000027	0.000027	5.8593	97.66%	
V*WP	1	0.000031	0.000031	6.8211	98.47%	
V*P	1	0.000022	0.000022	4.9114	96.36%	
WP* P	1	0.000015	0.000015	3.3633	92.09%	
WP*P*V	1	0.000016	0.000016	3.6305	93.12%	
Residual	24	0.000109	0.000005			

The resultant material was then investigated with an optical microscope to see what type of material was produced. It was observed that on the cases with low weight percent solutions fibers with intermittent beads were being created and on higher weight percent solutions non-beaded fibers were being created. As testing continued, it was observed during testing that constant pressure did not relate to a constant flow rate of a polymer solution from a syringe. The pneumatic cylinder and I/P transducer were replaced with a syringe pump (KD Scientific 780100) to better hold the syringe and control the flow of the polymer solution during testing. Results of electrospun material seen from the optical microscope are shown in Figure 6.



(A) (B)

Figure 6: Optical microscope images at 50x magnification. a) 4wt% PEO solution b) 8wt% PEO Solution.

Continued electrospinning results showed 4wt% PEO solutions only created beaded fibers. A review of journal articles showed no paper of electrospinning pure fibers with 4wt% PEO solutions. Testing became adjusted to see if all weight percents of PEO solution could be electrospun into pure fibers, and if Weber number help explain the difference between beaded and non-beaded fibers.

To create non beaded fibers for all weight percents tested, PEO solutions were held in either a 1-ml or a 10-ml syringe, based on spinning rate, and placed on a syringe pump (KD Scientific 780100). The syringe was fitted with a 23-gauge needle with an outside diameter of 0.62 – 0.64 mm and an inner diameter of 0.32 - 0.34 mm. A high-voltage power supply (Glassman High Voltage, Inc.) with a range of 0 to 30 kV was used to apply a potential between the syringe needle and the collector plate. A digital microscope (DinoCapture) was used to observe the electrospun jet and other phenomena during the electrospinning process.

A flat copper collector plate was placed 125 mm from the needle tip and was attached to the ground source. Voltages ranging from 3.5 kV to 11 kV were applied to the syringe via an alligator electronic connector clamped onto the needle. A LabView program was written to control the high-voltage power supply remotely. The testing at voltages of 5 kV and below required a 5 second pulse of 11 kV to produce a Taylor cone. The voltage was then reduced to the target value for the remainder of the test. This startup procedure was needed to overcome the surface tension of the solution, which would have kept electrospinning from occurring at lower voltages. During testing at lower voltages, the rate at which the PEO solution was ejected from the syringe was set at 0.02 ml/hr to ensure the Taylor cone stayed in the same conical shape throughout the test. Higher voltages, ranging from 6 to11 kV, did not require an initial pulse; they were able to be spun immediately after the voltage was introduced. The rate at which the syringe pump was operated, for these voltages, varied between 0.2 ml/hr to 1.5 ml/hr to ensure the stability of the Taylor cone. All tests were run for 10 to 20 minutes. During testing it was important to maintain steady flow conditions; this was done by making sure that none of the solution built up into large droplets on the needle and fall from the tip. This entailed several preliminary test runs of the electrospinning setup to determine the proper dispensing rate of the syringe pump.

An ultra-high molecular weight polyethylene film was placed in front of the copper plate to collect the fibers produced during testing. To determine the flow parameters such as flow rate and velocity, it was necessary to determine the mass of PEO deposited at the collector plate. Mass was measured with two methods before choosing the most efficient in the following manner. The syringe was weighed before and after the test was run. This mass difference was then compared to the mass difference of the polyethylene film from before and after the test, accounting for the appropriate proportion of water lost through evaporation. These two estimates of deposited mass were consistently within ten percent of each other, ensuring that the vast majority of PEO was being deposited on the polyethylene film. Immediately after electrospinning, the polyethylene sheet was placed on a shelf to dry for 24 hours. After drying, the sheets were weighed, then either sputtered coated with gold/palladium and taken to a JEOL 6400 Scanning Electron Microscope (SEM), or taken to an optical microscope and imaged with 50x magnification to measure the diameter of the spun fibers.

3.3 Results

3.3.1 Surface tension results

The surface tension of various PEO concentrations is shown in figure 7. It was observed that surface tension decreased as weight percent of the solution increased. The data agree well with Dietzel's values of surface tension of PEO solutions [22].

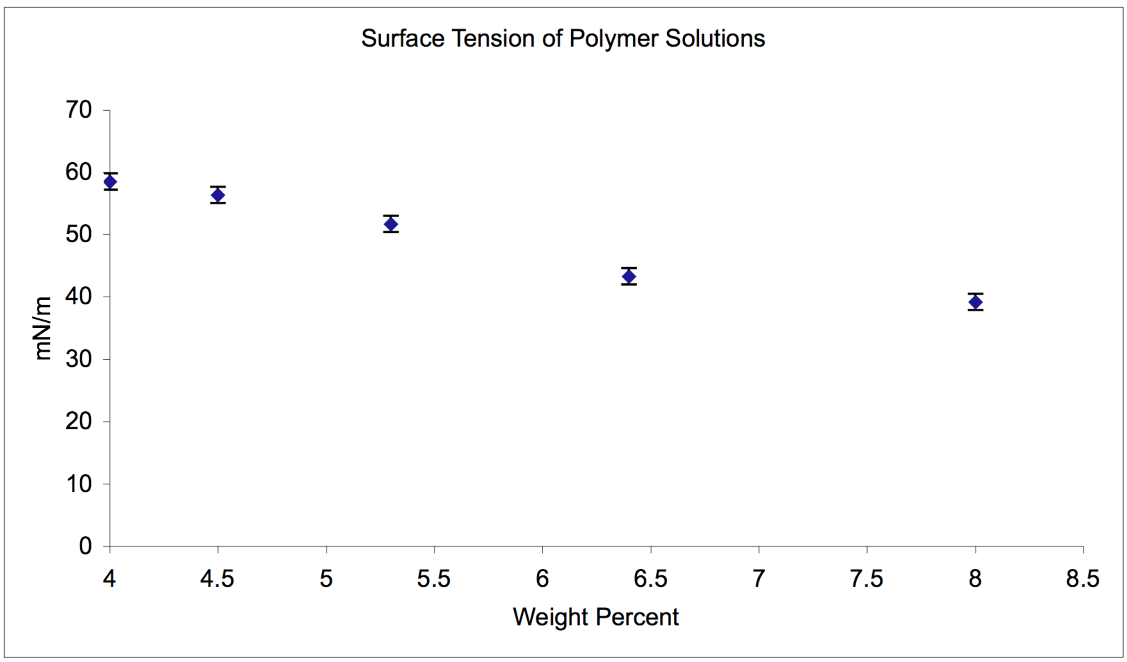


Figure 7: Graph of surface tension versus weight percent of the PEO solutions.

3.3.2 Fiber diameter

The electrospun fibers were taken to the SEM in order to get a detailed look at the resultant fiber morphology and to measure fiber diameters. The accelerating voltage for the SEM was set to 15 kV with a working distance of 15 mm. High magnification images were taken to measure the diameter of the electrospun material.

Because fiber diameter was used to determine the jet velocity (through a mass flow rate relation), the presence of entrained beads required a technique that would account for a representative material volume to be consistent with the non-beaded fibers. The diameter of fibers connecting two beads was very small, leading to artificially high velocities to be calculated. On the other hand, using the maximum diameter of the beads themselves resulted in very low calculated velocities. It was decided that a weighted average diameter must be used, which incorporated the diameters of both the connecting fibers and the beads in such a way as to maintain consistency across the various samples with entrained beads. Electrospun material that had entrained beads had connecting fiber diameters in the range of 80 to 150 nm and the beads themselves had diameters in the range of 400 nm to 1 μm. An average diameter of the resultant beaded fiber was calculated to effectively handle the varying diameter due to the entrained beads. By using the SEM images, a weighted average of fiber/bead diameter was calculated by measuring the ratio of connector fiber and bead lengths and assigning a corresponding weight to the diameter measurements. Averaged diameters of the beaded fibers were in the range of 280 to 350 nm in size, larger than the size of the fiber but smaller than the size of the beads.

3.3.3 Weber number

Mass flow rate was calculated by dividing the mass of PEO deposited onto the polyethylene film by the length of time the test was run. From mass flow rate, volume flow rate was calculated by dividing the mass flow rate by the calculated density of the polymer solution.

The velocity of the jet (U) was calculated by dividing the volume flow rate with the area of the jet before it reached the collection plate. In this study, the diameter of the jet near the end of the plate was assumed to closely approximate the diameter of the resultant fiber deposited at the target, which was imaged in the SEM. The measured diameter, as described above, was used to calculate the area of the jet. It was assumed that the liquid jet was cylindrical in shape as it traveled to the plate. Once velocity of the jet was obtained, Weber number was calculated based on (1).

In cases when the PEO material was spun into fibers with no beads present, the Weber number ranged from 0.12 to approximately 25, with velocities of the jet ranging from 15 to 50 meters per second. The SEM images in Figures 8 through 10 show different weight percents of PEO solution electrospun at different voltages, resulting in various fiber morphologies.

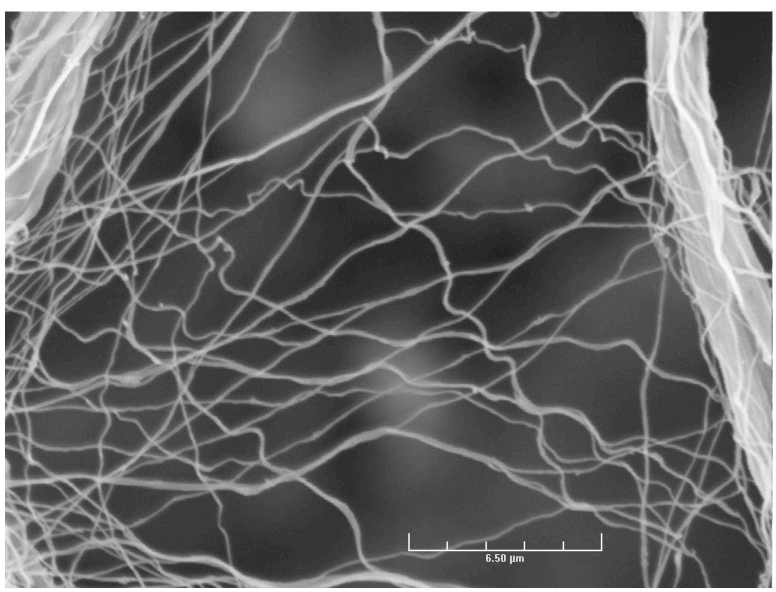
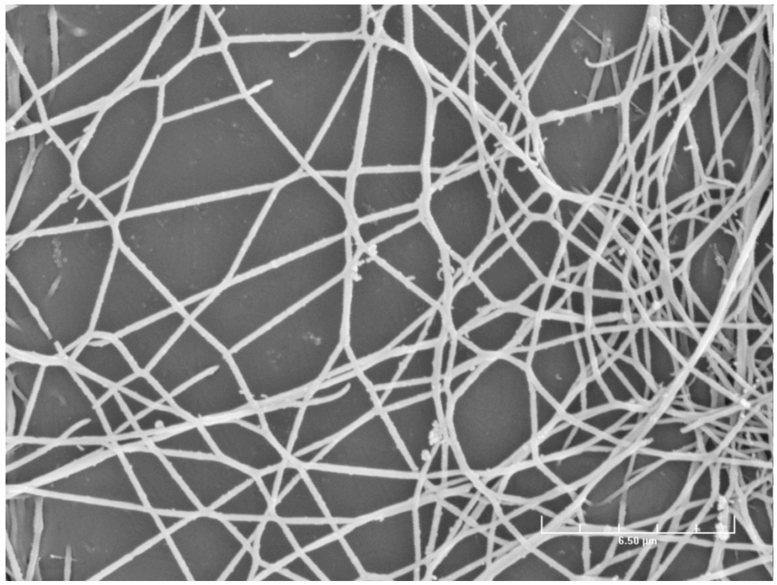
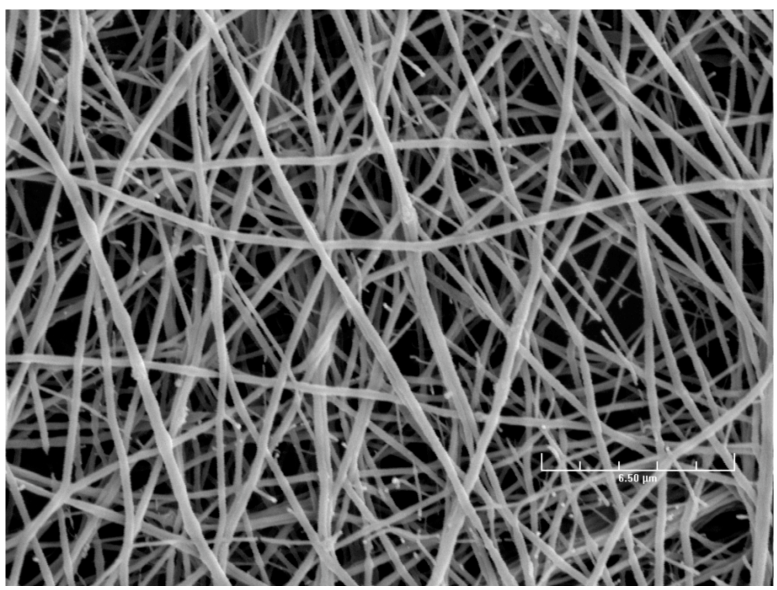


Figure 8: Electrospinning results of 4.5 kV voltage and 4wt% PEO solution.

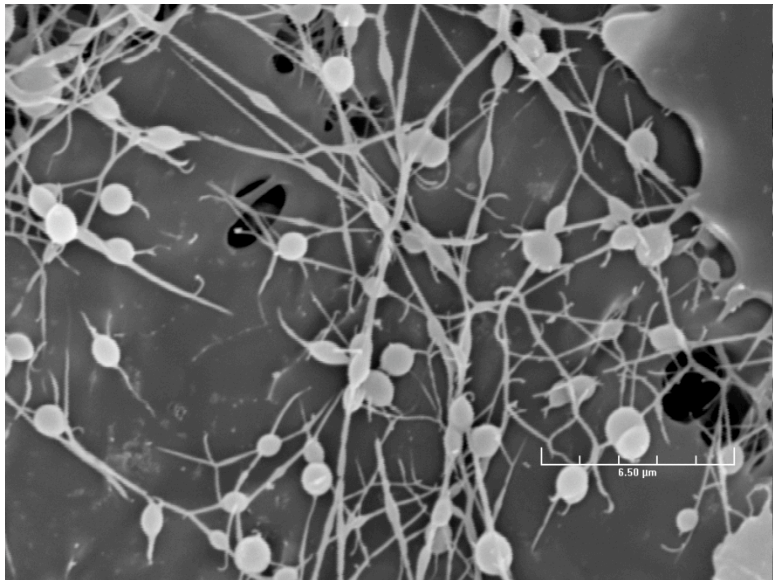


(A)

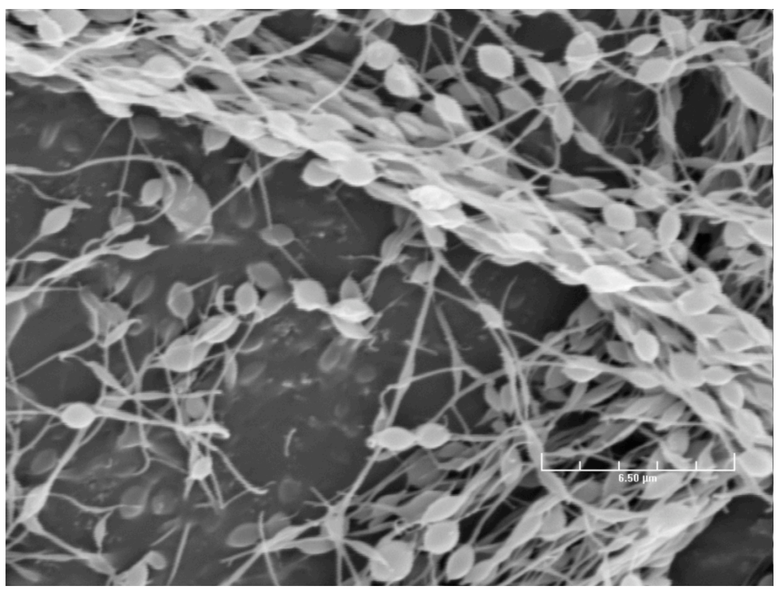


(B)

Figure 9: Electrospinning results for 5.3 wt% and 8 wt% PEO solution electrospun at a) 5.3 wt% 7 kV and b) 8wt% 10 kV.



(A)



(B)

Figure 10: Electrospinning results of 4wt% PEO/ water electrospun at a) 11 kV b) 6.8 kV.

In cases when the calculated Weber number was greater than approximately 40, morphologies with many entrained beads were observed. The velocities in these cases were calculated to be above 50 m/s. Table 1 tabulates Weber numbers calculated in this study and indicates that there is a transition from pure fibers to entrained beaded-fibers. It may be argued that this transition occurs between the approximate values of 26 and 40.

3.3.4 Statistical analysis and sorting the data

The mean and standard deviation of the fiber diameter was based on the number of test runs for each setting and the multiple diameters measured on each individual test run. The standard deviation for beaded fibers was much greater than the standard deviation for material with pure fibers, this is due to the differences in diameter of the beads and fibers. The standard deviation of beaded fibers was based on measurements of both beaded and fiber sections of the electrospun material. The standard deviation of fiber diameter can be seen in Table 4. The standard deviation of Weber number was calculated based on the measured values of: fiber diameter, mass flow rate, surface tension, and density. The equation to calculate standard deviation for Weber number can be seen below (2)

$$S_{Wb} = \sqrt{\left(\frac{\partial Wb}{\partial \dot{m}} * S_{\dot{m}}\right)^2 + \left(\frac{\partial Wb}{\partial d} * S_d\right)^2 + \left(\frac{\partial Wb}{\partial \rho} * S_\rho\right)^2 + \left(\frac{\partial Wb}{\partial \sigma} * S_\sigma\right)^2} \qquad (2)$$

The highest standard deviations calculated in Weber number, shown in Table 4, are when the electrospun material resulted in beaded fibers. This large variation in the standard deviation of Weber number is attributed to a combination of both the diameter of the material and the mass flow rate of the polymer solution**.** In (3), the calculation for

Weber number is rewritten for measured values. Notice in particular that the diameter d is cubed while the mass flow rate $\dot{m}$ is squared, causing these two factors to have a greater impact in the Weber number standard deviation. In the case of beaded fibers, both of these measurements have extremely high individual standard deviations. As a result, the standard deviation of Weber number for beaded fibers is also high.

$$Wb = \frac{16\dot{m}^2}{\sigma \rho d^3 \pi^2} \qquad (3)$$

When the experimental data is sorted with respect to voltage no clear indication can be seen when fiber or beaded fiber are created as well. The beaded fibers results can be seen on both ends of the data presented in the table 4.

Table 4: Electrospinning results sorted to voltage

Voltage (kV)	**Weight Percent**	**Diameter (nm)**		**Weber Number**		**Fiber or beaded fibers**
		Mean	$\mathbf{S_d}$	**Mean**	$\mathbf{S_{wb}}$	
4.5	4.0	147	29	2	1	fibers
6.9	4.0	198	51	50	40	beaded
7.0	5.3	260	10	6	0	fibers
7.0	6.6	220	20	3	2	fibers
7.0	8.0	170	10	26	5	fibers
10.0	4.0	318	310	71	210	beaded
10.0	5.3	205	55	75	140	beaded
10.0	6.6	240	40	14	4	fibers
10.0	8.0	295	55	13	6	fibers

When the same experimental data collected is sorted in terms of weight percent, the beaded fibers are grouped closely together but there is no clear indication that can be made on the parameters chosen that controls the morphology of fibers and beaded fibers electrospun. The results can be seen in the table 5.

Table 5: Electrospinning results sorted to weight percent.

Weight Percent	Voltage (kV)	Diameter (nm)		Weber Number		Fiber or beaded fibers
		Mean	S_d	Mean	S_{wb}	
4.0	4.5	147	29	2	1	fibers
4.0	6.9	198	51	50	40	beaded
4.0	10.0	318	310	72	210	beaded
5.3	7.0	260	10	7	0	fibers
5.3	10.0	205	55	75	140	beaded
6.6	7.0	220	20	4	2	fibers
6.6	10.0	240	40	8	4	fibers
8.0	7.0	170	10	6	5	fibers
8.0	10.0	295	55	11	6	fibers

When the experimental data is sorted according to the calculated Weber number, a clear separation between beaded and non-beaded fibers can be seen. No other correlations can be seen in voltage, weight percent, and diameter on the same table. Results are shown in Table 6.

Table 6: Electrospinning results sorted to Weber number

Weber Number		Fiber or beaded fibers	Voltage (kV)	Weight Percent	Diameter (nm)	
Mean	S_{wb}				Mean	S_d
2	1	fibers	4.5	4.0	147	29
3	2	fibers	7.0	6.6	220	20
6	0	fibers	7.0	5.3	260	10
13	6	fibers	10.0	8.0	295	55
14	4	fibers	10.0	6.6	240	40
26	5	fibers	7.0	8.0	170	10
50	40	beaded	6.9	4.0	198	51
71	210	beaded	10.0	4.0	318	310
75	140	beaded	10.0	5.3	205	55

3.4 Discussion

3.4.1 Surface tension

The surface tension of the PEO solution decreased with the increase in weight percent of PEO. While this may seem somewhat counterintuitive, this behavior has been demonstrated previously [22]. The drop in surface tension is due to the hydrophobic nature of the CH_2CH_2 segments of the PEO backbone ($-CH_2CH_2O-$). As the concentration of the polymer increases, the resulting solution thus becomes less and less hydrophilic [31]. As is currently hypothesized in this work, Weber number is a strong indicator of electrospun fiber morphology. Because the surface tension term is so prominent in the calculation of this dimensionless parameter, it becomes vital to understand the surface tension behavior of PEO and other polymer solutions. Most polymers in a solvent approach a critical concentration past which further polymer does not dissolve [31]. Therefore, the control of nanofibers morphology by using Weber must take into account the ability to change the surface tension of various polymer solutions.

3.4.2 Weber number

These results show that the Weber number can likely be used as an indicator of fiber morphology. Examination of Table 6 and Figures 8 through 10, show that there is a clear distinction in the Weber numbers that correspond to non-beaded and beaded morphologies. When Weber number exceeds approximately 40, all of the tests produced fibers with entrained beads. An informative exercise is to examine all of the 4 wt% PEO solutions studied. Previous researchers have reported the presence of entrained beads at low solution concentrations [10, 22], and the same was observed during this study at voltage settings in the same range as higher concentration solutions. However, the viscosity of the lower concentration solution is low and thus the extensional flow behavior of the electrospun jet allows for higher flow velocities and thus higher Weber values as shown in the table. When the voltage is reduced in order to reduce the flow velocity, entrained beads are no longer present and Weber values are correspondingly low. This same behavior was reproduced with a 5.3 wt% solution as shown in the table. The prediction and eventual control of fiber morphology based on Weber number must take in to account the process parameters that are controllable in a given electrospinning setup. In this case, the two directly controllable parameters were applied voltage and PEO concentration. An effect on all of the terms of Weber number could be observed from manipulation of these two parameters. Changing PEO concentration affected the density and surface tension of the solution, while applied voltage affected velocity of the jet. Furthermore, though not reported here, variation of PEO concentration affected extensional viscosity of the jet and thus greatly affected the resulting fiber diameter. The manipulation of these two process parameters, polymer concentration and applied voltage, is in line with other researchers working the electrospinning of polymer fibers.

As stated above, Weber number brings together various materials and process characteristics and incorporates them into one dimensionless number. It is envisioned that Weber number analysis could be applied to other polymer/solvent systems to control the nanofiber morphology. The transition range identified in this work for aqueous solutions of PEO, between 14 and 40, may not be the same for every polymer/solvent system tested, but this work shows that the concept of using Weber number to describe the free jet flow of electrospun fibers is valid. It follows that a transition range will exist in other systems such that Weber values below the transition will result in fibers while values above the transition will result in entrained beads. Future work is needed to identify the numerical values of these transitions in other polymer solutions.

The Weber transition range identified in this study differs from Lin's values for the first stage of fluid jet breakup, above approximately 45. The four stages of jet breakup in Lin's study include: Rayleigh breakup, first wind induced, second wind induced, and atomization [11]. It is hypothesized due to the resulting morphologies and calculated Weber numbers that this work involved only Rayleigh breakup of the jet. Further study would be needed to determine if the PEO solution is capable of other types of breakup, thus making additional morphology options available such as ultra-fine electrospun beads.

3.4.3 Sorting the data

Table 4 shows experimental results sorted with respect to voltage. The voltage used to electrospin PEO solutions strongly affects the velocity of the jet. The data does not show a strong correlation between voltage and the production of beaded fibers and non-beaded fibers. An interesting look at table 4 shows that at 10kV, the diameter of the fiber initially decreases then increases as the weight percent increases. In other studies, the diameter of the material increases as the weight percent increases [11, 16]. The experimental data shown in table 4 would indicate that it could also decrease; however, in this study, the method of measuring the diameter was different between the non-beaded fibers and the beaded fibers, which makes it impossible to declare any correlations between experimental data

When the same experimental data is sorted with respect to weight percent, as seen in table 5, there is not a clear indication of the difference between beaded fibers and non-beaded fibers. This is due to the fact weight percent does not account for certain operating parameters such as voltage. The weight percent of a polymer solution affects density and surface tension; these factors are important factors for determining Weber number but other material properties are important as well. The weight percent does not have a strong correlation with the velocity of the polymer jet or the morphology of the electrospun material.

The same experimental data in Table 6, separated with respect to Weber number, shows a separation between beaded fibers and non-beaded fibers. As indicated earlier, Weber number takes elements of both voltage (velocity) and weight percent (density and surface tension) to produce a unitless number to describe the electrospinning process. This suggests that both the voltage and weight percent are needed to predict the fiber morphology of electrospun material.

3.5 Conclusion

The following conclusions are made based on the results of this work:

Two fiber morphologies were observed in the electrospinning of PEO solution, pure fibers and fibers with entrained beads. The appearance of entrained beads was due to a mechanism of fluid-jet breakup.

Weber number is an appropriate gauge of the free-jet flow conditions of the electrospun jet, and thus can indicate the conditions that lead to the two morphology types. At Weber numbers below approximately 25, non-beaded fibers result, while at values above 40, entrained beads are produced.

The Weber values that indicate a transition from non-beaded to beaded fibers agree reasonably well with work done in the jet breakup of pure liquids. This signifies that fundamental free-jet breakup mechanics can describe the electrospinning process well.

4. INCORPORATION OF ELECTROSPINNING INTO AN UNDERGRADUATE ENGINEERING CURRICULUM

Electrospinning is an innovative material fabrication process that can provide useful learning experiences for undergraduate students in engineering and other scientific fields of study. Many different disciplines are involved in electrospinning, including physics, material science, fluid mechanics, dynamics and chemistry, to name several. Because of this richness in content, an instructor can use different research domains as a basis to relate theoretical problems to real world applications. Teaching a semester-long course focused on electrospinning will also allow students to see the cutting edge in nano-material fabrication, while helping them understand the foundation and basic principals of engineering, problem solving, and applying the scientific method.

4.1 Hierarchies of Learning

Prescribed learning outcomes for students in a semester course of electrospinning can advantageously be based on the New Taxonomy of Educational Objectives (NTEO). The New Taxonomy of Educational Objectives is a refinement of the learning outcomes proposed in Bloom's taxonomy. Bloom's Taxonomy was developed in the 1950s and gauges the levels of learning a student exhibits based on a six-level system [32]. The six levels of Bloom's Taxonomy are: knowledge, comprehension, application, analysis, synthesis, and evaluation. Knowledge is a student's ability to acquire information; this can be done through recognition or recall. Comprehension is a student's ability to understand information through different forms of communication, either spoken or inscribed. Application is the ability to use knowledge in problems without being guided through the solution. Analysis is the application and comprehension of information in an organized manner. Synthesis is the creation of new knowledge structures. An example of synthesis is when a person uses physics principals to explain the motion of the soccer ball or the curve of a baseball. It is primarily the use of knowledge in ways unrelated to the original context in which the information was encountered. Evaluation is the ability to make decisions about the usefulness of the knowledge acquired. In practical terms, this means the students would have the ability to critically assess the value of what they are learning. They understand the material well enough to accept or discard the new information given to them.

The NTEO uses similar categories to Bloom's taxonomy but in more detailed fashion [32]. The NTEO-based taxonomy has three domains of knowledge, with each domain of knowledge consisting of six levels of processing.

The three domains are:

- Information
- Mental procedure
- Psychomotor procedure

Information is defined in the NTEO as general knowledge, such as facts and definitions. Mental procedure is the ability to employ formulas and procedures for problems that require multiple steps to identify a solution. Psychomotor procedure is the mental memory of the information concerning a movement the body has made, like playing basketball or performing an advanced surgery.

Within the above domains are levels of processing. These include:

- Retrieval
- Comprehension
- Analysis
- Knowledge utilization
- Metacognition
- Self-system processing.

Retrieval is the process of querying permanent memory in order to provide details to working memory in order to perform a task. Working memory is data that is processed based on the stimulation of the senses [32]. Working memory helps a person differentiate hot from cold, true from false, and safe versus hazardous senses that are experienced continually. Comprehension is the transfer of knowledge from the working memory into the permanent memory. One of the issues with this cognitive process is that in most cases, the transfer from working memory to permanent memory changes the original meaning of the acquired knowledge and is thus somewhat dependent on the context in which the knowledge was encountered. Analysis is the elaboration of the knowledge, such as the identification of new meanings for terms already developed. Knowledge utilization is the collection of cognitive processes that are used to accomplish a task. Metacognition is the control and acknowledgement of all thought and specifically addresses the evaluation and management of knowledge as a task is performed. Self-system processing is the integration of attitude, emotion, and beliefs that determine a student's willingness to focus attention to learn. The proposed course in electrospinning will naturally exercise a number of these cognitive learning levels;

however, attention will be paid specifically to addressing the mental procedure domain at the level of knowledge utilization.

4.2 Learning Outcomes

The overall goal for the proposed electrospinning course is to impart in the students the ability to describe the process, identify its key components, and to suggest methods of implementing it for engineering applications. The act of pursuing these outcomes will entail a number of pedagogical techniques as well as various subject areas. With regards to the NTEO, these learning outcomes fall into the knowledge utilization level in the mental procedure domain.

To ensure the overall objectives are met by the end of the semester, assessments will be used to judge the students' attainment of the learning outcomes. These assessments will include conventional examinations, laboratory activities, peer-teaching projects, and homework. The activities that are proposed to prepare students for these assessments will address the NTEO domains of information and mental procedure, and various sublevels within these domains. Laboratory activities will be used throughout the semester to develop students understanding of the different electrospinning concepts and their interrelationships; it will also help students understand learning outcomes being taught in the lecture. Such activities are intended to address the comprehension level in the mental procedure domain of the NTEO. Student directed lectures and presentations, on the other hand, would address the comprehension level of the knowledge domain of the NTEO. Homework will assess students' week-to-week understanding, while exams will evaluates the students understanding as a whole.

4.3 Course Information

It is intended that this course would be technical elective offered at the senior level due to the course scope and material being covered. The prerequisites for the course are chemistry, physics, and engineering.

Studies have shown that students will retain the information better when they are offered the material in a interactive environment [33]. This means the lectures in the class should vary from in class exercises, discussions, lectures, open ended questions, and other forms of learning through out the semester.

During lab time, the class will be split into four person groups to run experiments; this will increase the amount of equipment and space needed to electrospin material. However, the small group environment will encourage teamwork among the students. In Springer's work small groups employs a more cooperative learning environment than competitive or individual learning environment [34]. Students will have a higher enjoyment of the class and will also retain information more effectively working in small groups.

The exception will be that any student, regardless of background, will be able both to document the steps required to obtain an electrospun product, as well as having the ability to recreate others' experiments.

Homework assignments given will vary from problem solving situations and journal article reading throughout the semester. The homework will make sure that students are studying the material outside the classroom lectures. The test questions given to the students throughout the semester will vary from short answer, critical thinking, matching, or true and false questions depending on the material covered in the lectures and homework assignments given. The variety of test questions should allow an accurate evaluation of how well the students understand the learning outcomes set for the course [35].

Most of the information the students will use during the semester will be based on widely available technical information, such as U.S. patents, as well as experimental details and results published by researchers in the technical literature. A significant portion of the students' time will be spent in locating and critiquing references. Due to the young age of this research area, there is not an established textbook that could be used in a course such as this. This is also partially due to the fact that the material integrates many engineering and scientific disciplines. Excerpts from textbooks in subjects of various fields like: electrical engineering, physics, fluid mechanics, and safety precautions manuals will be used as reference material to validate equations and theories used in the electrospinning process.

Grading for the course will be based on the performance of various assessments that are tied to the course learning outcomes. These assessments will be designed to get a holistic measurement of the acquired knowledge during and at the conclusion of the course.

4.4 Course Instruction

The course will be split into three five-week sections that will each focus on a particular topic; these areas include polymer science, physics and dynamics, and fluid mechanics. A schedule of presentation of course topics is shown in Table 7. The subdivision of the course in this way allows the instructor to set up specific learning objectives that builds into the overall course outcomes. Furthermore, this structure allows for the assessment and any required correction in a single subject area before moving to a very different focus. By establishing the proficiency of the students' learning in each of the three course subdivisions, the instructor will have some assurance that the overall outcomes will be achieved by the course conclusion.

Table 7: Proposed semester schedule.

	Topic	Activities and Assessments
Week 1	Introduction into electrospinning	Read journal articles about electrospinning
Week 2	Polymers: Properties	Stress Strain question with polymers
Week 3	Polymer: Chemistry and Processing	Drawing out Polymer Chemical structures
Week 4	Polymer Chemistry and Processing cont'	Relating Polymer Structures with density, and elastic modulus
Week 5	Exam review, Exam	Draw and label the different thermoset and thermoplastic polymers.
Week 6	Physics of Electrospinning	Calculate the surface tension of the polymer solution
Week 7	Voltage relationship with Physics	Calculate the force generated by the voltage
Week 8	Polymer Properties based on chemical Structure	Determine the viscosity of the polymer solutions
Week 9	Taylor Cone, Electrically Driven Jet	Journal Articles
Week 10	Exam Review, Exam	Determine the volume flow rate and the mass flow rate of the polymer jet
Week 11	Velocity of the Jet	Calculate the velocity of the Liquid jet
Week 12	Whipping Phenomenon	Journal Articles
Week 13	Modeling the Spinning Process	Journal Articles
Week 14	Weber Number calculations	Calculate the Weber number of different electrospun material
Week 15	Exam Review, Final Lab	

4.5 The First Five Weeks

The first five weeks of the course will focus on polymer science. The specific learning outcomes for the first five weeks are:

- The ability to recognize and construct different polymer chemical structures
- The ability to determine the effects of various polymer chemical structures on mechanical properties
- The ability to understand the rate at which technology develops

The first learning outcome falls within the comprehension and retrieval levels of the information domain in the New Taxonomy. It is important that students recognize the fundamental details of polymer structures, and how various polymer structures are different from each other. The second outcome falls into the comprehension and analysis levels of the knowledge domain in the NTEO. Accordingly, the students should also be able to identify the manner in which particular chemical agents will react with different polymers, the relative biocompatibility of various polymers, and the effect of weight percent of polymer in solution on material properties. It is also necessary that students develop the ability to determine the effect of polymer structure on various mechanical properties. The third outcome falls into the retrieval level of the knowledge domain of NTEO. Electrospinning is fairly new in the vocabulary for researchers and scientists, and has only seen a surge in study over the past ten years. However, electrospinning was first patented in the 1930s [1]. Students must recognize that technological advances typically come from years of scientific study and may not have practical applications for decades.

The learning outcomes for the first five weeks of class will be assessed in five homework assignments, two lab reports, and one exam. The homework assignments will measure the students' week-to-week understanding of the lectures and in-class activities. The homework will primarily focus on the drawing of polymer chemical structures and the development of the electrospinning process through reading journal articles. The experiments will assess students' capacity to work on a team, to perform tasks with multiple steps, and to relate experimental data to topics covered in the lectures. An exam will be the final assessment of the students' understanding of the learning outcomes covered in the lecture, homework, and experiments. The exam will have at least one question that will test a students understanding of the learning outcomes desired from the NTEO criteria for the first five weeks of class.

One way for students to achieve the learning outcomes in the first five weeks is to have activities where the students are required to draw polymer structures in a peer-support setting, such as on a whiteboard in the classroom. Having the students themselves draw in the classroom fits with Felder's work on using interactive methods to improve student understanding scientific material [2]. Other activities that will aid the students in achieving the learning outcomes are in-class exercises. A proposed classroom exercise is to have students calculate the molecular weight and density of various polymers, and tabulate these results with reported mechanical properties. This exercise would help students understand the correlation between structures and properties.

The experiments performed in the first five weeks will be used as a means to introduce the concepts of polymer solubility and the behavior of polymer solutions. The students will perform the task of melting the polymer and also dissolving the polymer into solution, and using each of the liquids in molding into a specified solid shape by cooling or by solvent casting. These polymers will be thermoplastics in order to show students the reversibility of polymers and that they are unlike metals. Thermosets and epoxies are not a focus of class experiments, but will be briefly explained in class lecture. The second experiment will be about the mechanical properties of various polymer classes. Different polymers with the same molecular weight, length and diameter will be put under similar tensile strength tests to illustrate the variability in properties that results based on polymer chemical structure.

4.6 Second Five Weeks

The specific learning outcomes from the second five weeks of the course, focusing on physics and dynamics of electrospinning are:

- The ability to investigate complex problems and construct equations that describe the electrospinning process and help explain the creation of polymer liquid to a polymer fiber.
- The ability to categorize important parameters in electrospinning
- The ability to make good decisions regarding ones safety when operating the electrospinning setup

The first learning outcome is associated with the knowledge utilization level of the mental procedure domain of NTEO. Students should be able to draw free body diagrams and be able to compute the proper units of measurement based on laws of gravity, conservation of motion, conservation of mass, and others used in describing dynamics of the electrospinning process. One example the students will see throughout the second five weeks is the conversion of an applied voltage (volts) to a force (newton) through the electro-physical models of forces acting on a charge. The second learning outcome is associated with the analysis level of the information domain in NTEO. Categorizing the different parameters of electrospinning and the different resultant materials, students need to understand the material morphologies created by various processing parameters when electrospinning. The third learning outcome comes from the knowledge utilization level of the information domain. Some of the most significant hazards in electrospinning deal with the high voltage needed to spin the material and the solvents used to create the polymer solutions. Students must be able to make the proper decisions regarding their safety and their fellow students' safety as well.

The learning outcomes will be assessed with five homework activities, two laboratory experiments, and one examination. The homework will again be used to measure the students' problem solving skills on a weekly basis. It will also introduce students to integrating topics discussed in the first five weeks with the new topics being covered in the second five weeks. The experiments will assess students' judgment regarding safety as well as the ability to follow detailed laboratory procedures. The exam will be composed of eighty percent of material covered in the second five weeks of lecture and twenty percent of material from the first five weeks of lecture. The exam will also be used to determine the achievement of the learning outcomes.

For students to achieve the learning outcomes, lectures will have problems that introduce students to free body diagrams and other methods of problem solving. An example will be to predict the velocity of an object that is thrown or to predict the distance a ball will travel after rolling down a hill. These problems will introduce students' velocity, force, acceleration, and distance, which are important factors to the electrospinning process. Some lectures will focus on the conversion of applied voltage to force in order to help students understand how the electrospun polymer jet is pulled to the collector. Students will also be introduced to the development and mechanics of the Taylor Cone, which can be seen when fiber is beginning to be pulled from the nozzle tip, as well as how voltage and other gravitational forces affect this phenomenon. A Taylor cone is a conical shape of polymer solution that forms when the force created by the applied electric field is sufficient to draw polymer solution to a point at which the fiber is drawn. Figure 11 is the look of the Taylor cone from a digital microscope; a small string can be seen as the polymer is being pulled to the plate.

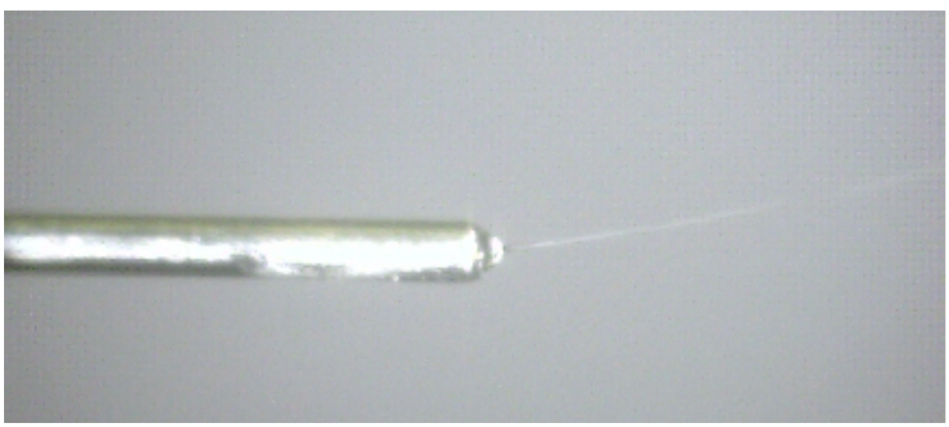


Figure 11: Taylor Cone from digital microscope of PEO/water solution.

Example problems will also be used extensively in the course and will be used to generate student discussion during lecture sessions.

A proposed experiment will illustrate how the applied voltage develops the Taylor cone and eventually pulls the polymer to the plate. This experiment will correlate to the material covered in parts of the lecture. Another experiment will employ different polymer solutions, and require students to measure the viscosity and surface tension of the solutions and electrospin them with the same voltage. This experiment will reintroduce concepts learned in the first five weeks of the class in order to integrate the earlier material.

4.7 Third Five Weeks

The specific learning outcomes from the third five weeks of the course, focusing on the theories and phenomena when electrospinning are:

- The ability to investigate and integrate previously reported research on electrospinning
- The ability to recognize and characterize phenomena and other theories associated with electrospinning
- The ability to manipulate various electrospinning parameters to create polymer nanofibers and assess the results

The first learning outcome fits into the comprehension and knowledge utilization levels of the information domain in the NTEO. Students must be able to demonstrate that they can understand what other researchers are investigating in the field of electrospinning. The second learning outcome addresses the analysis level of mental procedure in the NTEO. Students must be able to propose reasons for differences in electrospinning results. The third learning outcome is in the knowledge utilization level of the mental procedure domain in the NTEO. The students must demonstrate that they can propose particular electrospinning parameter settings in order to produce desired result.

The learning outcomes will be assessed with four homework assignments, two experiments, and a final laboratory exercise. The homework assignments will focus heavily on literature review and analysis in order to determine student comprehension of the work done by other researchers. The experiments will assess the students' ability to classify and match theories and other phenomenon with the results of the electrospun material. The final laboratory exercise, which will be the same as a final exam, will assess the students' complete understanding of the material learned over the entire course.

The final five weeks of the course will be more discussion based than the previous subdivisions. It is proposed that the classroom will be arranged in a fashion that encourages open discussion among the students on topics brought up by the instructor. Experiments in the final weeks will focus on calculation of the velocity of the electrospinning jet under various conditions. This experiment will tie together all of the topics covered in the previous ten weeks of lecture. Another experiment will challenge the students to recreate an experiment from a selected journal article that was discussed in class. This activity will highlight to the students that their results may be different from another researcher, and enable them to make suggestions about the causes of differences. Furthermore, it will put students in a position to critically analyze their own, and the previous, results.

The final laboratory exercise will be an overview of all course material with an emphasis on the velocities of the electrically driven jet as well as the diameters of the resultant fibers. Students will produce two different sets of polymer nanofibers with diameters varying in size through electrospinning.

4.8 Conclusion

The course material and the manner of which classroom lectures and experiments are conducted are intended to excite students about electrospinning as well as engineering in general. Studies have shown that courses where the lecture format was varied and the students were made to actively participate in lectures resulted in better grades for the students, higher interest in more advanced degrees in engineering, and a more positive attitude towards engineering [33]. The proposed electrospinning course is intended to motivate students to become better engineers and researchers, and to increase the likelihood that they will choose to pursue graduate degrees in engineering. Also, because so many different fields of study can be used to help students fully understand the electrospinning process that learning about electrospinning will allow students to see that all fields of study are important, no matter what field or sub-field of study students want to major in.

5. CONCLUSIONS

The use of Weber number as an indicator when beaded or non-beaded fibers are created, is a step in the better understanding of the electrospinning process. Using the different weight percents of PEO solution and electrospinning the solutions at different voltages and rates, the creation of nanofibers of varying diameters and surface morphologies were observed. Calculating the Weber number of the polymer jet shows that beaded and non-beaded fibers have a distinct difference, and the calculation of Weber number can be used to better control the electrospinning process. The difference can be seen in the 4wt% PEO solution, where on very low voltages non-beaded fibers were electrospun and on high voltages beaded fibers were electrospun. While the calculation of Weber number will not have the same cut off points for every polymer solution used in electrospinning, it can still be used to understand the type of breakup occurs to the jet as it travels to the plate, as well as understanding the surface morphology of electrospun fibers. A better understanding of the electrospinning process will lead to more discoveries for future applications of the material properties the nanofibers have.

Teaching electrospinning in a semester course enables an instructor to relate experimental data and apply it principals of basic engineering. It also allows for multiple disciplines like physics, mathematics, engineering, and material science be combined into one field of study. Electrospinning as a class also allows the instructor to teach in multiple different formats because of the differences in disciplines and styles, which will give the instructor the opportunity to teach visually, experimentally, lectures, and through discussion. The course should give students a look at the cutting edge of nano material research and will also sparks the student's interest in pursuing more advanced engineering degrees.

The simplicity of the electrospinning process allows for both avenues to be explored.

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APPENDIX A

Many earlier test done with electrospinning revealed interesting images that lead to the study of fluid mechanic principals to describe polymer jet breakup. Images of other methods employed through out the work can be seen in the following figures shown below. Images of Figure 12 are from earlier test when the researchers understood about electrospinning and what changes can occur when varying the spinning parameters. Pictures below have either a different weight percent of PEO solution or have a different voltage applied to them.

Images in Figure 13 and 14 are PEO material looked under the optical microscope. The optical microscope images were taken at 50x zoom. The images help us see the beads or pure fibers created from the electrospinning process. However the measuring of the diameters proved to be very difficult due to the scale of measurement and the size of the fibers, which led to use with the SEM.

The images in Figures 15 and 16 are more SEM images of the electrospun material. The SEM images taken during the study ranged in magnification. The 10000x images were too blurry to get an accurate diameter measurement of the fiber. The 1000x images were to far away to measure diameter accurately but gave a clear description of how the electrospun fibers looked and joined together.

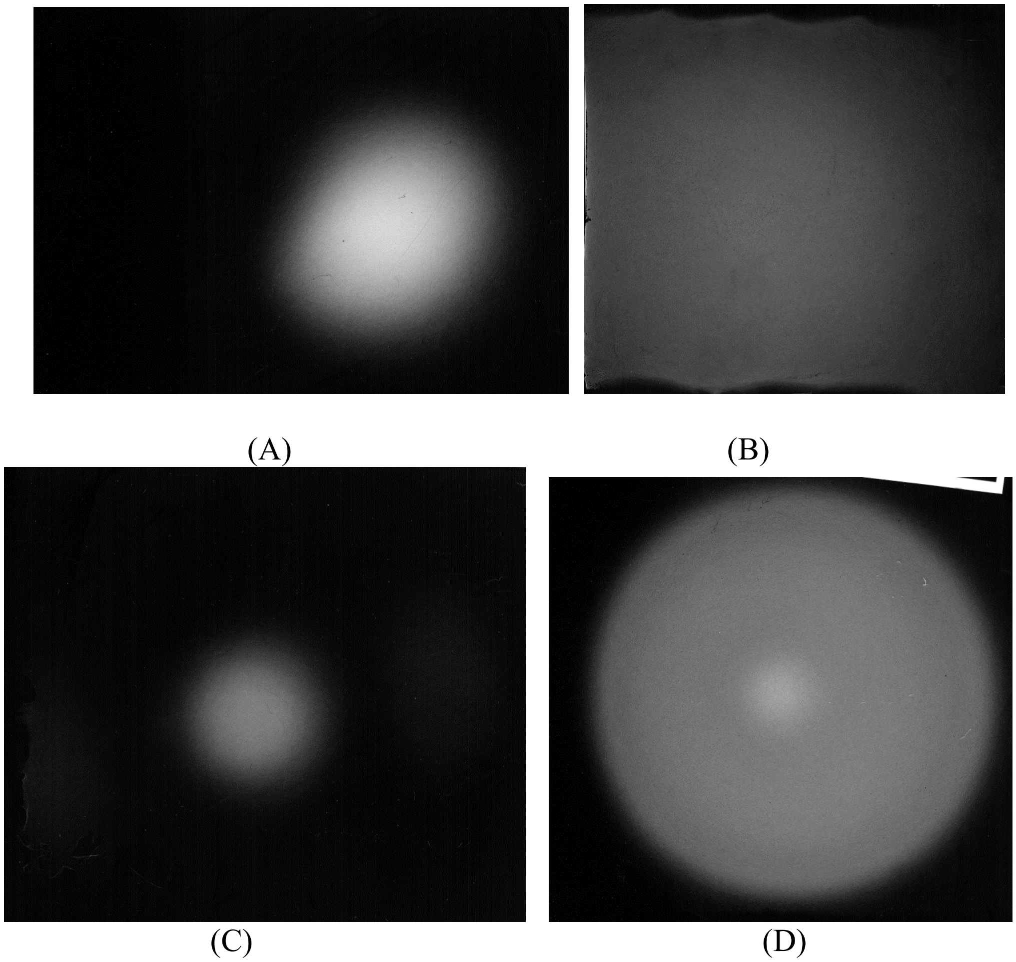
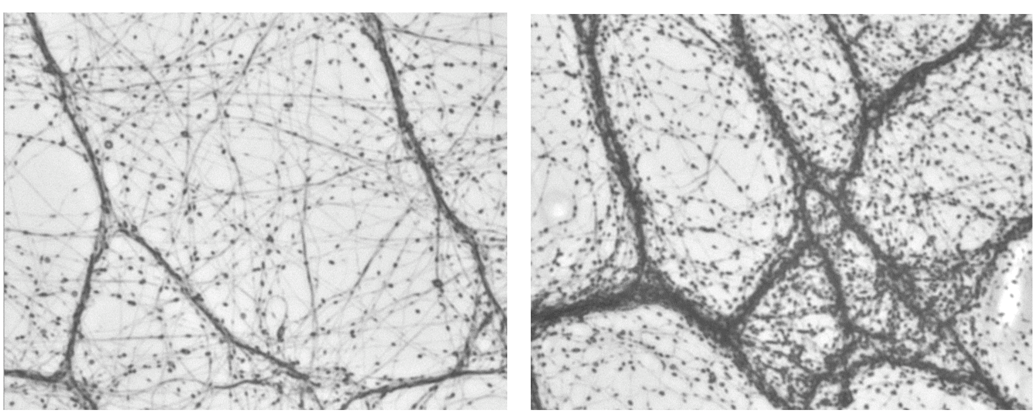
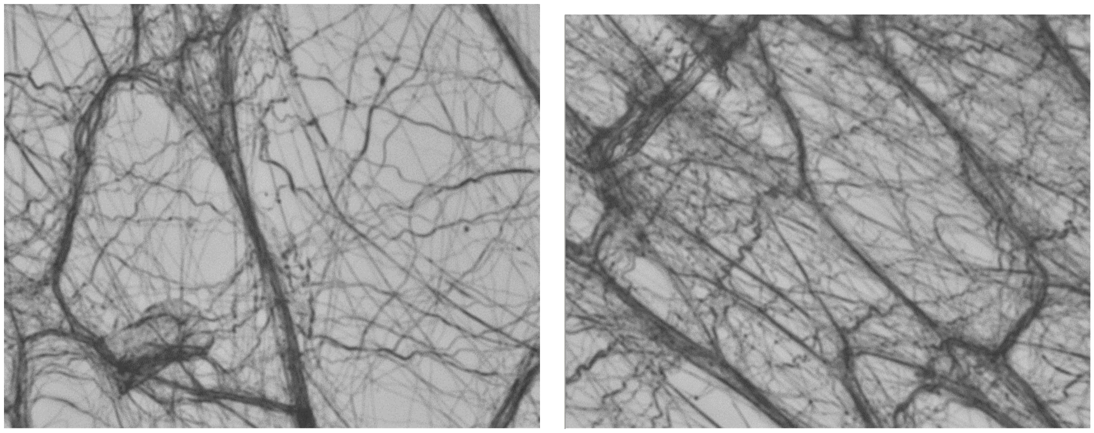


Figure 12: Scanned images of electrospun fibers and the shapes made a) 8wt % PEO at 10 kV b) 4wt% PEO at 7kV c) 8wt% PEO at 7kV d) 4wt% PEO at 10kV.



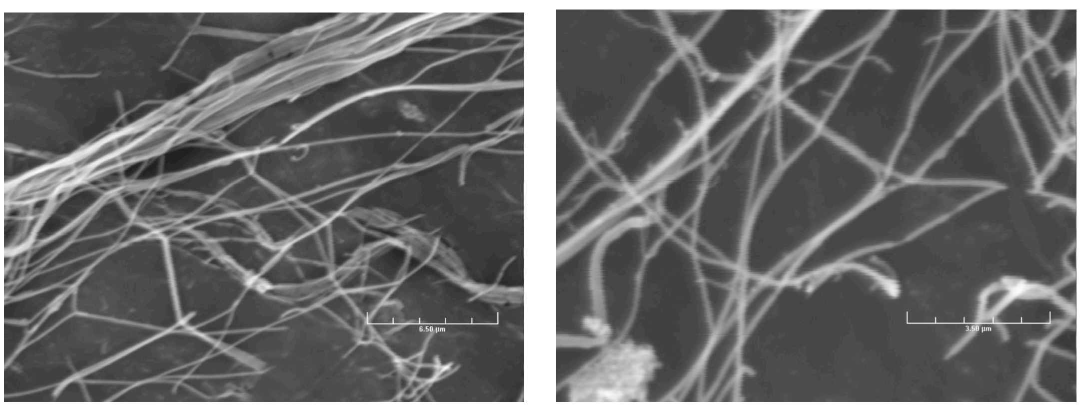
(A) (B)

Figure 13: Images of beaded fibers from the optical microscope at 50x zoom a) 4wt% PEO at 7kV b) 4wt% PEO at 10kV.



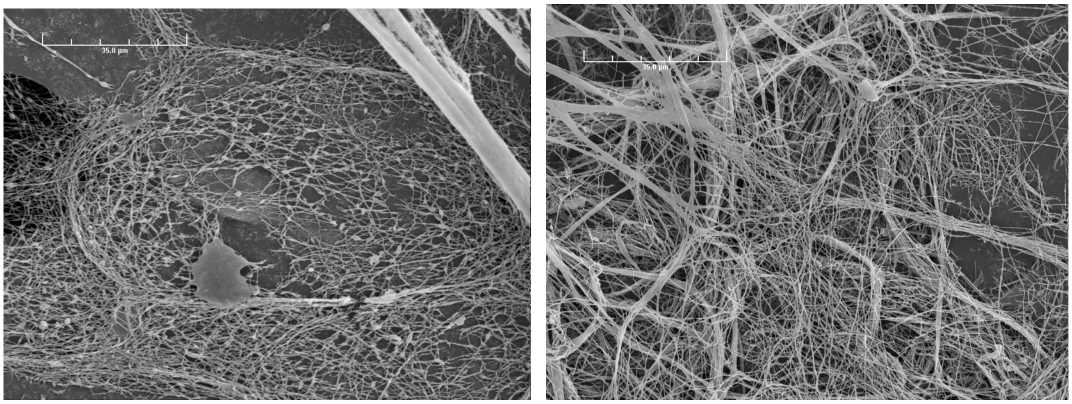
(A) (B)

Figure 14: Images of non-beaded fibers from the optical microscope at 50x zoom a) 8wt% PEO at 10kV b) 8wt% PEO at 10kV.



(A) (B)

Figure 15: SEM images of electrospun material at high magnification a) 8wt% PEO b) 6.6wt% PEO.



(A) (B)

Figure 16: SEM images of electrospun material at low magnification a) 4wt% PEO b) 6.6 wt% PEO.

APPENDIX B

Syllabus
Electrospinning Course MEEN/MEMA 435

Course Description: Electrospinning is a technique used to create polymer or ceramic material on the nanoscale. To create nanomaterial through electrospinning, many different engineering and scientific disciplines are used to describe the process. This course will explain the main components of electrospinning with a mix of laboratory exercises to show how electrospinning works with the theories and principals associated with the electrospinning. *Students are expected by the end of the semester to understand the key components of electrospinning and should be able to recreate the process on their own.*

Textbooks: No Textbook is required for the class. Students are expected to print out the journal articles and other online documentation for classroom discussion when advised to.

Supplemental Material: Material is not required for students to read but is strongly encouraged. Instructor will notify students about course material throughout the semester

Meeting Times: MWF 2 pm -2:50 pm

Instructor: Dr Christopher Call
Email: ccall83@tamu.edu
Office Hours: TR 12-2pm, or by appointment

Attendance: Attendance to the course is not mandatory, but is strongly encouraged if the student wants to receive a good score on test and homework assignments. If class is missed, it is the student's responsibility to find out the work missed from other classmate. He may not go to the professor.

Grading Policy:
Exam 1: 15%
Exam 2: 15%
Final Exam (Lab): 15%
Labs: 35%
Homework: 20%

Schedule:

	Topic	Activities and Assessments
Week 1	Introduction into electrospinning	Read journal articles about electrospinning
Week 2	Polymers: Properties	Stress Strain question with polymers
Week 3	Polymer: Chemistry and Processing	Drawing out Polymer Chemical structures
Week 4	Polymer Chemistry and Processing cont'	Relating Polymer Structures with density, and elastic modulus
Week 5	Exam review, Exam	Draw and label the different thermoset and thermoplastic polymers.
Week 6	Physics of Electrospinning	Calculate the surface tension of the polymer solution
Week 7	Voltage relationship with Physics	Calculate the force generated by the voltage
Week 8	Polymer Properties based on chemical Structure	Determine the viscosity of the polymer solutions
Week 9	Taylor Cone, Electrically Driven Jet	Journal Articles
Week 10	Exam Review, Exam	Determine the volume flow rate and the mass flow rate of the polymer jet
Week 11	Velocity of the Jet	Calculate the velocity of the Liquid jet
Week 12	Whipping Phenomenon	Journal Articles
Week 13	Modeling the Spinning Process	Journal Articles
Week 14	Weber Number calculations	Calculate the Weber number of different electrospun material
Week 15	Exam Review, Final Lab	

American with Disabilities Act (ADA) Policy Statement

The American with Disabilities Act (ADA) is a federal discrimination statute that provides comprehensive civil right protection for persons with disabilities. If you have a disability that requires accommodation, Contact Services for Student with Disabilities

Aggie Honor Code:

An aggie does not lie, cheat, or steal or tolerate those who do.

Members caught cheating will be turned into the university and disciplinary actions will be taken that can lead to expulsion from the university.

VITA

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